

## CHEMICAL PROCESSES

**Focus:** This lab consists of three experiments: one related to fluid instabilities which can affect environmental systems, or chemical processing operations; and two related to chemical separation processes.

### **Part 1: Salt Fingering**

#### **Overview:**

This is primarily an observational experiment. You will produce two-component, stably-stratified solutions in each of two containers. (A solution is stably stratified when the lighter component is above the heavier component.) In each of the containers the upper and lower solutions will have specific gravities of 1.025 and 1.050, respectively. In both cases the lower solution will be a water/salt solution tinted with food coloring. The upper solutions will be different. One will be a water/sugar solution, the other a water/salt solution. In the containers provided, you will produce stably-stratified layers with the tinted solution on the bottom and the clear solution on top. But, to create these layered solutions without mixing, requires a special technique.

#### **Procedure:**

First, half-fill the containers with a clear solution--one each with water/sugar and with water/salt (These are the less dense solutions). Next, pour about a half inch of the colored salt solution into a wash bottle whose nozzle has been outfitted with a very fine Teflon tube. Remove all air bubbles by gently squeezing the wash bottle (you should only lose only a few drops of solution). CAREFULLY place the tube in the container so that so that no tinted solution leaves the wash bottle until the tip of the tube is almost on the bottom. VERY SLOWLY squeeze out the contents of the wash bottle. Since this colored solution is the heavier of the two, it should stay at the bottom. If you're careful, you should have a sharp line of demarcation between the clear and tinted solutions.

In the container with the two densities of water/salt solutions, observe what happens over about ten minutes. Do the same with the water/salt and water/sugar solutions. Something different is happening. With different solutes, diffusion can be a complicated process. Salt diffuses more rapidly in water than sugar. As a result, the upper solution eventually becomes more dense than the lower one creating an instability. "Fingers" of the colored salt solution begin to ascend into the upper layer, carrying with them the food coloring. Meanwhile, fingers of the clear sugar solution migrate downward to replace the heavier fluid. Here, mixing consists of both convection and diffusion.

**Write-up:**

This is essentially an observation lab--describe the two experiments and what you see. Would it work for very large or very small density differences? How long does it take for the salt fingers to penetrate to the top? Do they reach the very top? Try to estimate the average distance between the fingers.

**Part 2: Paper Chromatography****Overview:**

Chromatography separates different chemical substances—in this case colored dyes--by making use of their different flow rates while being transported by a diffusing solvent. Flow rates are defined as being the distance a solute (a chemical substance) travels divided by the distance traveled by the solvent. For example, one color in a solvent may move farther than another color in the same solvent even if they are given the same length of time to move.

If the end of a piece of absorbent paper with an ink pen line on it is placed in solvent, the solvent will be drawn up the surface of the paper by capillary action. As it passes the ink line, the ink will dissolve in the solvent and be carried along with the solvent (this is mass transport). The ink, though, will be differentially retarded by the paper. A competition for ink is set up between the solvent and the paper, and as a result, the ink will travel up the surface of the paper more slowly than the solvent. The relative strength of the ink's attraction for the paper and solvent varies between inks. If there is more than one ink in a given pen, each ink will travel at its own pace, and you will see each separate ink emerge. In addition, some solvents will dissolve certain inks better than others. When solvents are used that do not dissolve the ink well, most of the ink will be left behind at the original line and only a small amount will travel up the paper.

**Procedure:**

You will be given three pieces of filter paper (a coffee filter could have been used) that you should cut into rectangles about 4cm x 10 cm. Using the three pens provided, make a line segment about 0.75cm long with each of the pens about 1 cm from one end of the each paper strip. The distance between marks should be about 0.75cm; the end result should be a dashed line where each line segment has been made with a different pen. Make multiple passes so the ink is dark. Put several lines of the same color directly on top of each other rather than overlapping them. The objective is to provide sources of dye which can diffuse up the length of the strips. Let the ink dry while you make a horizontal pencil mark connecting the two ink lines and extending beyond them. Make another pencil mark near the other end of the strip. These will mark the end point of the experiment.

You will be conducting nine separate experiments: three with water as a solvent, three with methanol(POISON) as a solvent, and three with 2-propanol(POISON). Each of the ink marks is its own separate experiment. First pour solvent into the developing tank to a depth of about 1 cm. Attach the plain end of your prepared paper strip to a straightened paper clip. This wire will be used to suspend the filter paper over the solvent. Lower the paper into the solvent to a depth of 2-3mm, but not so deep that the ink marks are in the solvent. Cover the tanks with a piece of paper to minimize evaporation.

The solvent will rise, partially carrying the ink. Once the solvent reaches the pencil mark (which won't dissolve in the solvents), remove the paper strip from the tank and let it dry. Measure the farthest excursions of all ink lines on this "chromatogram". The ratio of the distance traveled by the ink to the distance traveled by the solvent is the flow rate for that ink in that solvent. Perform this experiment with water, methanol, and 2-propanol as solvents.

### **Write-up:**

First, describe what happened, taking care to differentiate between different colors and different solvents. For the methanol and 2-propanol experiments, calculate flow rates from the purple ink and present a graph showing the flow rate of each component as a function of the proportion of 2-propanol in the solvent (approximately 100% for 2-propanol and 0% for methanol). Assuming that flow rate changes linearly with 2-propanol concentration, draw a line which represents the expected flow rate for each component in various concentrations. Deduce the expected equations of each of these lines.

Notice that for the purple pen, one ink component flows faster in 2-propanol while the other flows faster in methanol. If we were attempting to separate these two components, and we were really unlucky by choosing the wrong concentration of methanol, we could obtain no separation. Estimate the methanol concentration in 2-propanol of this most undesirable solvent?

## **Part 3: Distillation**

### **Overview:**

The objective of this experiment is to apply the process of distillation to a mixture of ethanol and water in order to obtain a new mixture with an increased concentration of ethanol. In addition, the results of the experiment will be used to approximate the equilibrium relationship between ethanol and water.

Distillation has been described as the "work-horse" of chemical engineering because of its widespread use in industry. Distillation is a type of separation process that is useful in separating components of a liquid mixture that have different boiling points.

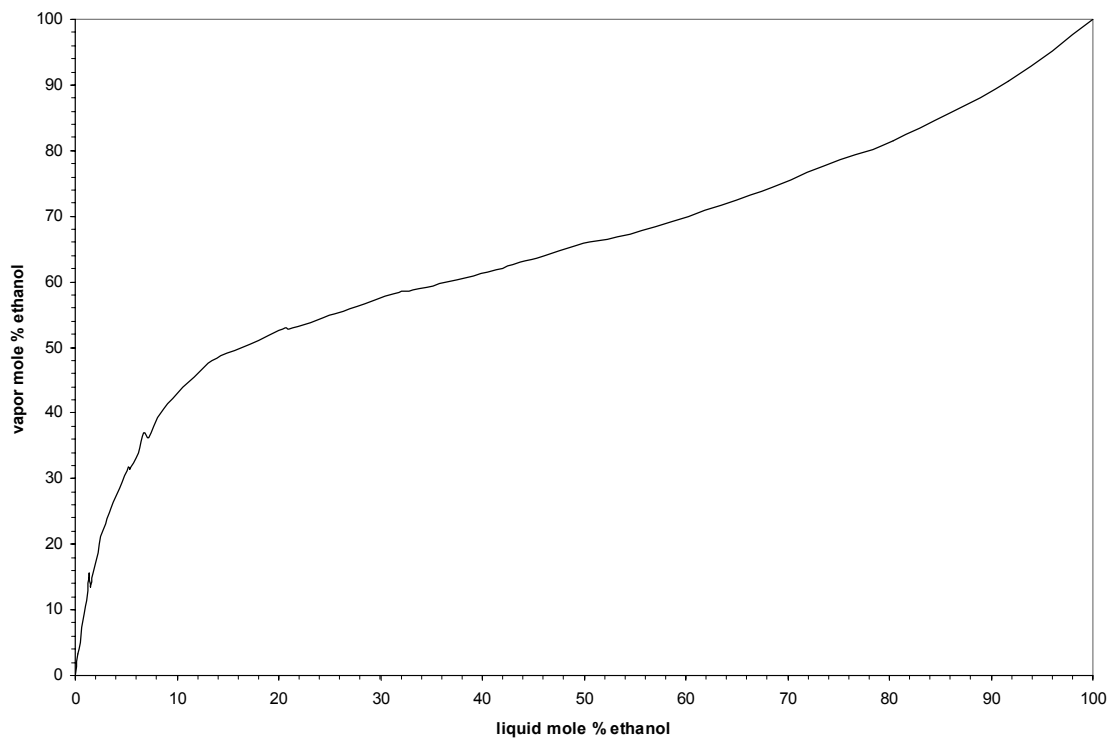
For example, consider a mixture consisting of two components, A and B, that have boiling points of 70°C and 100°C, respectively. When the mixture begins to boil, the vapor phase that is formed will be richer in A than the liquid phase. This is because A has a lower boiling point and vaporizes more easily than B. Therefore, A has a greater tendency to enter the vapor phase while B tends to remain in the liquid phase. Of course, if the entire mixture were allowed to boil away, the resulting vapor would have the same concentration of A and B as the original liquid mixture. However, if only a fraction of the liquid is allowed to boil, the vapor will contain a higher concentration of A than the original liquid mixture.

As the mixture continues to boil, the compositions of both the vapor and liquid phases change with time. Since A enters the vapor phase more quickly than B, the concentration of A in the liquid phase decreases while that of B increases. Also, as the temperature of the boiling liquid increases, more B vaporizes as time passes, and the concentration of B in the vapor phase also increases with time.

If some fraction of the original mixture vaporizes and the vapor is collected and allowed to cool and condense in a separate container, the new liquid mixture will have a higher concentration of A than the original did. With each repetition of this process, we would obtain a new liquid mixture with a higher A concentration than the previous mixture. This is basically how distillation works. It is just a series of vaporization and condensation processes that continues until a desired concentration is reached.

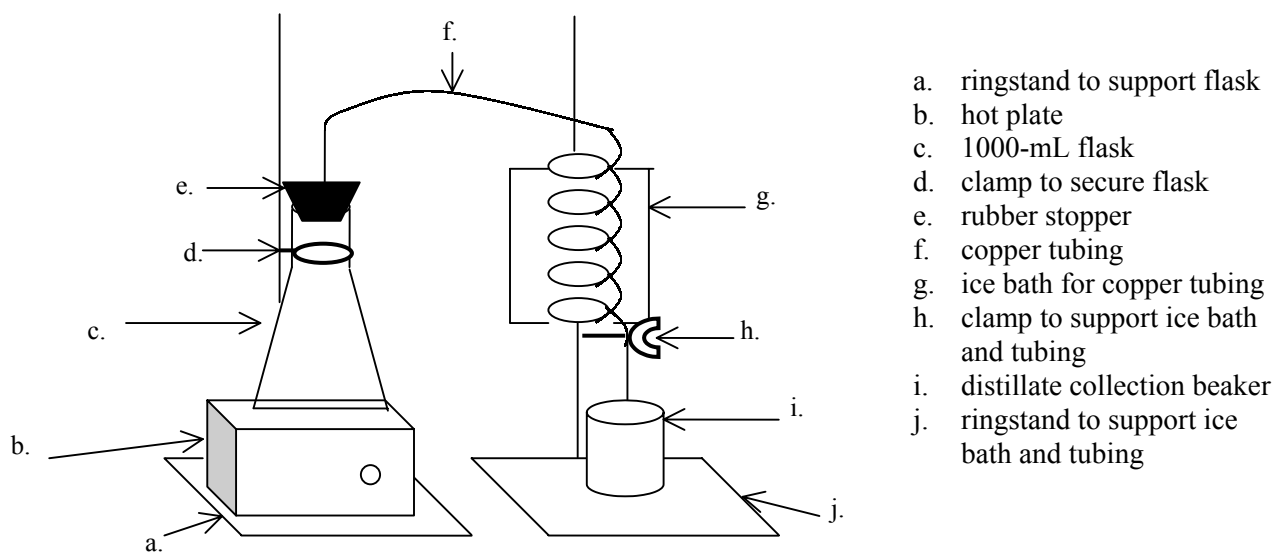
In general, an equilibrium stage in any type of unit operation process is defined as an area in which different phases are brought into close contact so that a component can be redistributed between the phases to equilibrium concentrations. In the simple batch distillation setup in this experiment, liquid in the 1000-mL flask is in close contact with the vapor phase that forms as the mixture boils, and this is the only place where this situation is present in the setup. Therefore, there is one equilibrium stage in this batch distillation. Assuming everything works ideally, the vapor phase is in equilibrium with the liquid in the flask at any given time.

The type of equilibrium information that is needed for analyzing this separation process is that which shows the vapor-liquid equilibrium relationship for an ethanol/water mixture. The most useful representation of the data is a y versus x graph, where y and x are the vapor and liquid phase concentrations, respectively, of the more volatile component, ethanol. Points on the curve depicted in such a graph represent the concentration of ethanol in the vapor and liquid phases at equilibrium. Along the equilibrium curve, pressure is constant, but temperature is different at each point. A picture of the ethanol/water y versus x graph is shown below.



### Procedure:

This experiment is a small-scale batch distillation. The basic setup is shown below.



The original ethanol/water solution is placed in the 1000-mL flask. Heat to the mixture is provided by a hot plate. A rubber stopper caps the flask, and it contains a small hole through which a piece of copper tubing is inserted. The long piece of tubing coils downward through an ice bath, and its purpose is to carry vapors away from the flask and allow them to cool and condense to the liquid phase. (The condensed liquid is referred to as *distillate*.) The condensed liquid empties into a beaker at the bottom of the coil.

During the experiment, you will allow the flask mixture to boil. Vapors produced from boiling will escape the flask through the copper tubing and will condense in the cooled, coiled part of the tubing forming the distillate. The distillate will drain into the collection beaker. Three 40-mL samples of distillate will be collected. In order to analyze this process quantitatively, it is necessary to know the concentration of ethanol in the distillate and in the flask mixture at the beginning and end of the experiment.

In order to determine the percent of ethanol in the various liquid volumes, a temperature / specific gravity / mass fraction correlation chart for the ethanol/water system in Perry's Chemical Engineers' Handbook is used. You will measure the specific gravity and temperature of a liquid and use these two pieces of data with the chart to determine the mass fraction of ethanol. There are five liquid samples for which you will need to determine the mass fraction of ethanol: the initial solution, three distillate samples, and the final mixture in the flask at the end of the experiment.

The specific gravity is measured using a hydrometer. To use the hydrometer, collect approximately 40 mL of liquid in a 50-mL graduated cylinder. Place the hydrometer in the liquid and give it a gentle push downward so that it bobs up and down. Once the hydrometer comes to a rest, read the specific gravity at the surface level of the liquid. When taking hydrometer readings, it is very important that the hydrometer is floating freely and not resting against the side of the graduated cylinder. Also, readings are very sensitive to temperature, so be sure to record the temperature of the liquid with each hydrometer reading.

Begin the experiment by preparing the initial 800-mL ethanol/water mixture. The mixture should be 10% (by volume) ethanol. There are two important rules to follow while preparing the mixture:

- 1) Do not use the markings on the flask to measure volumes; they are not very exact.
- 2) Measure the ethanol and water **separately** using a graduated cylinder. Do not mix ethanol and water together in the graduated cylinder while measuring the volumes for the initial mixture; mixing will affect the volume readings.

To make the solution, measure the appropriate volumes of each liquid using a graduated cylinder. Pour measured volumes of liquid from the cylinder to the flask, and keep track of the total volume of the liquid using the cylinder's markings.

After making the solution, carefully swirl the flask to ensure that the ethanol and water are mixed well. (stir instead?) Now, measure the temperature and specific gravity of the initial solution. While doing this, be very careful not to lose any of your solution as this will affect your final calculations.

After determining the specific gravity and temperature of the initial solution, carefully pour the sample back into the flask, trying to be sure that none is lost. Place the rubber stopper (with the

copper tubing in it) on the flask. The tubing should stick out past the stopper approximately ½” into the flask.

Turn the hot plate setting to 9 and leave it at this setting for approximately 10 minutes. Then, turn the heat setting to 5 ½. The solution should not be boiling yet, but it will probably begin to boil within 8-10 minutes. Once the solution begins to boil, it should be about 3-5 minutes until you observe a distillate stream emptying into the collection beaker. Throughout the experiment, be sure that the flask mixture does not boil harshly.

After the beaker has collected approximately 40 mL of distillate, quickly replace it with an empty beaker. This needs to be done as rapidly as possible so that minimal distillate is lost. Cover and label the beaker containing the first distillate sample.

The temperature / specific gravity / mass fraction correlation chart only goes up to 40°C. Therefore, if the distillate is warmer than 40°C, you will need to allow it to cool before measuring its specific gravity. If it is already cool enough, it is a good idea to go ahead and measure its specific gravity while collecting the second distillate sample. To take the specific gravity measurement, pour the contents of the beaker into a 50-mL graduated cylinder. First, record the volume of the sample. This will be important for later calculations. (If you collected more than 50 mL of distillate, determine the volume using a larger graduated cylinder or measure the volume in sections with the 50-mL cylinder.) After recording the volume, use the 50-mL graduated cylinder and the hydrometer to determine the sample’s specific gravity. (If the cylinder is too full, some of the liquid can be poured out after the volume is measured since it is not necessary to have the total volume present to measure specific gravity; specific gravity is not dependent on volume.)

Use the same procedure to collect the second distillate sample. The third distillate sample is collected in a similar manner with a few variations towards the end of the experiment. Once approximately 40 mL has collected in the beaker, turn the hot plate off and remove it from under the flask. (Be sure that the clamp supporting the flask is secure!) Do not remove the distillate collection beaker until the mixture in the flask has stopped boiling for about 5 minutes. This helps to ensure that no vapors remain in the tubing.

After the distillate collection beaker has been removed, the flask can be removed from the clamp, and the stopper/tubing apparatus can be detached. However, be sure to keep the flask covered while it is cooling so that no vapors escape. The flask mixture will need to cool quite a bit before its specific gravity can be measured. (In the meantime, finish measuring the specific gravity of the three distillate samples.) A cool water bath will help expedite the cooling process of the flask mixture. In addition, gently swirling the liquid in the flask will help release heat more quickly.

### **Write-up:**

Chemical engineers typically perform mole balance analyses on processes as a means of checking to see that all material is accounted for. In general, this means checking to see that

$$\text{Input} + \text{Generation} - \text{Output} - \text{Consumption} = \text{Accumulation}$$

In this lab the mole balance can be thought of in terms of

$$\text{Moles in original flask mixture} = \text{Moles in Distillate 1} + \text{Moles in Distillate 2} + \text{Moles in Distillate 3} + \text{Moles in final flask mixture}$$

Mole balances can be carried out for the total material in the process or for individual components. From the measured volume, specific gravity, and mass fraction of the original mixture, distillate samples, and the final mixture, you are able to calculate the moles of ethanol and water in each of these volumes of liquid. Perform a mole balance for both the ethanol and water. If necessary, propose explanations for any discrepancies in the balances.

If the assumption is made that all of the vapor produced in the flask travels through the copper tubing and is collected as distillate, then the mole fraction of ethanol in the distillate sample is the same as that of the vapor in the flask. (Actually, this method is not exact. The 40-mL distillate samples are collected over a period of time during which the concentration of the vapor and distillate continue to change. Thus, the mole fractions of the distillate samples reflect a sort of time-averaged concentration rather than the concentration at an instantaneous moment in time.)

The equilibrium relationship between ethanol and water determines the concentration of the vapor and liquid phases. A liquid mixture with a certain concentration of ethanol will produce a vapor phase of a unique concentration, and these concentrations are predicted by the equilibrium relationship. In this experiment, the original mixture's concentration of ethanol is known, and the first distillate sample gives an approximation of the concentration of the corresponding vapor phase. This information makes it possible to plot a point on the ethanol/water equilibrium curve. Further, since it is known how much ethanol is removed from the flask in the first distillate sample, the new concentration of ethanol in the flask can be calculated. And, as before, the second distillate sample gives an approximation of the vapor phase concentration that corresponds to the new flask mixture concentration. The same rationale can be applied for the third distillate. Therefore, with the three distillate samples, three points on the equilibrium curve can be determined. Calculate values for these three points and plot them to construct an  $x/y$  ethanol/water equilibrium curve. Compare your curve with the given reference equilibrium curve. Suggest possibilities for any differences between the two.

Distillation Lab Data Sheet

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	Volume	Temperature	Specific Gravity	Mass fraction ethanol
Initial Flask Mixture				
1 <sup>st</sup> distillate sample				
2 <sup>nd</sup> distillate sample				
3 <sup>rd</sup> distillate sample				
Final flask mixture				

**Densities of ethyl alcohol (C<sub>2</sub>H<sub>5</sub>OH) in water**

(% alcohol by weight)

<b>%</b>	<b>10C</b>	<b>15C</b>	<b>20C</b>	<b>25C</b>	<b>30C</b>	<b>35C</b>	<b>40C</b>
<b>0</b>			0.99823	0.99708	0.99568	0.99406	0.99225
<b>1</b>			636	520	379	217	034
<b>2</b>			453	336	194	031	.98846
<b>3</b>			275	157	014	.98849	663
<b>4</b>			103	.98984	.98839	672	485
<b>5</b>	098	032	.98938	817	670	501	311
<b>6</b>	.98946	.98877	780	656	507	335	142
<b>7</b>	801	729	627	500	347	172	.97975
<b>8</b>	660	584	478	346	189	009	808
<b>9</b>	524	442	331	193	031	.97846	641
<b>10</b>	393	304	187	043	.97875	685	475
<b>11</b>	267	171	047	.97897	723	527	312
<b>12</b>	145	041	.97910	753	573	371	150
<b>13</b>	026	.97914	775	611	424	216	.96989
<b>14</b>	.97911	790	643	472	278	063	829
<b>15</b>	800	669	514	334	133	.96911	670
<b>16</b>	692	552	387	199	.96990	760	512
<b>17</b>	583	433	259	062	844	607	352
<b>18</b>	473	313	129	.96923	697	452	189
<b>19</b>	363	191	.96997	782	547	294	023
<b>20</b>	252	068	864	639	395	134	.95856
<b>21</b>	139	.96944	729	495	242	.95973	687
<b>22</b>	024	818	592	348	087	809	516
<b>23</b>	.96907	689	453	199	.95929	643	343
<b>24</b>	787	558	312	048	769	476	168
<b>25</b>	665	424	168	.95895	607	306	.94991
<b>26</b>	539	287	020	738	442	133	810
<b>27</b>	406	144	.95867	576	272	.94955	625
<b>28</b>	268	.95996	710	410	098	774	438
<b>29</b>	125	844	548	241	.949922	590	248
<b>30</b>	.95977	686	382	067	741	403	055
<b>31</b>	823	524	212	.94890	557	214	.93860
<b>32</b>	665	357	038	709	370	021	662
<b>33</b>	502	186	.94860	525	180	.93825	461
<b>34</b>	334	011	679	337	.93986	626	257

<b>%</b>	<b>10C</b>	<b>15C</b>	<b>20C</b>	<b>25C</b>	<b>30C</b>	<b>35C</b>	<b>40C</b>
<b>35</b>	162	.94832	494	146	790	425	051
<b>36</b>	.94986	650	306	.93952	591	221	.92843
<b>37</b>	805	464	114	756	390	016	634
<b>38</b>	620	273	.93919	556	186	.92808	422
<b>39</b>	431	079	720	353	.92979	597	208
<b>40</b>	238	.93882	518	148	770	385	.91992
<b>41</b>	042	682	314	.92940	558	170	774
<b>42</b>	.93842	478	107	729	344	.91952	554
<b>43</b>	639	271	.92897	516	128	733	332
<b>44</b>	433	062	685	301	.91910	513	108
<b>45</b>	226	.92852	472	085	692	291	.90884
<b>46</b>	017	640	257	.91868	472	069	660
<b>47</b>	.92806	426	041	649	250	.90845	434
<b>48</b>	593	211	.91823	429	028	621	207
<b>49</b>	379	.91995	604	208	.90805	396	.89979
<b>50</b>		0.91776	0.91384	0.90985	0.90580	0.90168	0.89750
<b>51</b>	.91943	555	160	760	353	.89940	519
<b>52</b>	723	333	.90936	534	125	710	288
<b>53</b>	502	110	711	307	.89896	479	056
<b>54</b>	279	.90885	485	079	667	248	.88823
<b>55</b>	055	659	258	.89850	437	016	589
<b>56</b>	.90831	433	031	621	206	.88784	356
<b>57</b>	607	207	.89803	392	.88975	552	122
<b>58</b>	381	.89980	574	162	744	319	.87888
<b>59</b>	154	752	344	.88931	512	085	653
<b>60</b>	.89927	523	113	699	278	.87851	417
<b>61</b>	698	293	.88882	446	044	615	180
<b>62</b>	468	062	650	233	.87809	379	.86943
<b>63</b>	237	.88830	417	.87998	574	142	705
<b>64</b>	006	597	183	763	337	.86905	466
<b>65</b>	.88774	364	.87948	527	100	667	227
<b>66</b>	541	130	713	291	.86863	429	.85987
<b>67</b>	308	.87895	477	054	625	190	747
<b>68</b>	074	660	241	.86817	387	.85950	407
<b>69</b>	.87839	424	004	579	148	710	266
<b>70</b>	602	187	.86766	340	.85908	470	025
<b>71</b>	365	.86949	527	100	667	228	.84783
<b>72</b>	127	710	287	.85859	426	.84986	540
<b>73</b>	.6888	470	047	618	184	743	297
<b>74</b>	648	229	.85806	376	.84941	500	053
<b>75</b>	408	.85988	564	134	698	257	.83809
<b>76</b>	168	747	322	.84891	455	013	564
<b>77</b>	.85927	505	079	647	221	.83768	319
<b>78</b>	685	262	.84835	403	.83966	523	074

<b>79</b>	442	018	590	158	720	277	.82827
<b>%</b>	<b>10C</b>	<b>15C</b>	<b>20C</b>	<b>25C</b>	<b>30C</b>	<b>35C</b>	<b>40C</b>
<b>80</b>	197	.84772	344	.83911	473	029	578
<b>81</b>	.4950	525	096	664	224	.82780	329
<b>82</b>	702	277	.83848	415	.82974	530	079
<b>83</b>	453	028	599	164	724	279	.81828
<b>84</b>	203	.83777	348	.82913	473	027	576
<b>85</b>	.83951	525	095	660	220	.81774	322
<b>86</b>	697	271	.82840	405	.81965	519	067
<b>87</b>	441	014	583	148	708	262	.80811
<b>88</b>	181	.82754	323	.81888	448	003	552
<b>89</b>	.82919	492	062	626	186	.80742	291
<b>90</b>	654	227	.81797	362	.80922	478	028
<b>91</b>	386	.81959	529	094	655	211	.79761
<b>92</b>	114	688	257	.80823	384	.79941	491
<b>93</b>	.81839	413	.80983	549	111	669	220
<b>94</b>	561	134	705	272	.79835	393	.78947
<b>95</b>	278	.80852	424	.79991	555	114	670
<b>96</b>	.80991	566	138	706	271	.78831	388
<b>97</b>	698	274	.79846	415	.78981	542	100
<b>98</b>	399	.79975	547	117	684	247	.77806
<b>99</b>	094	670	243	.78814	382	.77946	507
<b>100</b>	.79784	360	.78934	506	075	641	203